

(19)



(11)

EP 1 658 388 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:

21.05.2014 Bulletin 2014/21

(51) Int Cl.:

C22C 1/00 (2006.01)

(86) International application number:

PCT/US2004/027921

(21) Application number: **04817753.9**

(22) Date of filing: **27.08.2004**

(87) International publication number:

WO 2005/052198 (09.06.2005 Gazette 2005/23)

(54) **HIGH TEMPERATURE POWDER METALLURGY SUPERALLOY WITH ENHANCED FATIGUE CREEP RESISTANCE**

PULVERMETALLURGISCHE HOCHTEMPERATUR - SUPERLEGIERUNG MIT VERBESSERTER ERMÜDUNGS- UND KRIECHFESTIGKEIT

SUPERALLIAGE DE LA METALLURGIE DES POUDRES HAUTE TEMPERATURE AVEC LA RESISTANCE AMELIOREE A LA RUPTURE PAR FATIGUE ET FLUAGE

(84) Designated Contracting States:
DE GB

(30) Priority: **29.08.2003 US 651480**

(43) Date of publication of application:
24.05.2006 Bulletin 2006/21

(73) Proprietor: **Honeywell International Inc.**
Morristown, NJ 07960 (US)

(72) Inventors:
• **HIEBER, Andrew, F.**
Scottsdale, AZ 85260 (US)

• **MERRICK, Howard, F.**
Phoenix, AZ 85048 (US)

(74) Representative: **Buckley, Guy Julian et al**
Patent Outsourcing Limited
Cornerhouse
1 King Street
Bakewell
Derbyshire DE45 1EW (GB)

(56) References cited:
EP-A- 1 195 446 US-A- 4 388 124
US-A- 5 393 483 US-B1- 6 468 368
US-B1- 6 521 175

EP 1 658 388 B1

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

DescriptionBACKGROUND OF THE INVENTION

5 **[0001]** The present invention generally relates to a nickel based superalloy composition.

[0002] Nickel based superalloys have been extensively used in manufacturing gas turbine engine components. Gas turbine engines having hotter exhaust gases and which operate at higher temperatures are more efficient. To maximize the efficiency of gas turbine engines, attempts have been made to form gas turbine engine components, such as turbine discs, having higher operating temperature capabilities. In particular, there is considerable commercial interest in superalloys for turbine and compressor disk applications which exhibit strength and creep resistance at relatively high temperatures eg 704° C - 816° C (1300-1500° F), as well as resistance to fatigue crack initiation at the lower temperatures eg 260° C - 593° C (500-1100° F) often experienced in compressor and turbine disk bores. Higher temperature dwell crack growth resistance is also a significant parameter.

10 **[0003]** The previous generation of higher temperature capability disk alloys of the prior art are limited to about 649 - 704° C (1200-1300° F) operating temperature, and include such commercially used alloys as P/M Astroloy, Rene' 88 DT, and IN100. Such disk alloys, including the most recent generation of alloys, are typically made by inert gas atomization into powder form. The powder is subsequently screened to an appropriate size range and consolidated by hot compaction or by hot isostatic pressing (HIP). The consolidated powder is then extruded into a form suitable for isothermal forging into a shape that can be machined into an engine component. Components may also be formed by hot isostatic pressing (HIP) without the extrusion and isothermal forging steps, and subsequently machined to final shape. These methods of manufacture are common throughout the industry for high gamma prime volume fraction disk alloys.

20 **[0004]** US Patent No. 6,521,175 B1 to Mourer, et al. discloses a nickel based superalloy which contains 1.9 to 4.0 wt. % tungsten. The superalloy of Mourer, *et al.* sacrifices some low-temperature dwell fatigue crack growth performance to achieve improved creep performance.

25 **[0005]** As can be seen, there is a need for a nickel based superalloy composition which exhibits enhanced fatigue crack initiation life at temperatures of 260-593° C (500 to 1200° F), as well as enhanced resistance to creep at temperatures of 649-788° C (1200 to 1450° F). Dwell crack growth resistance at these higher temperatures of 649-788° C (1200 to 1450° F) is also of importance.

30 SUMMARY OF THE INVENTION

[0006] In the present invention, there is provided a nickel based superalloy composition, comprising: 16.75 to 17.25 weight % Co, 10.5 to 11.2 weight % Cr, 2.4 to 2.7 weight % Mo, 5.1 to 5.5 weight % W, 3.4 to 3.8 weight % Al, 3.6 to 4.0 weight % Ti, 1.3 to 1.7 weight % Ta, 0.85 to 1.15 weight % Nb, 0.02 to 0.04 weight % C, 0.025 to 0.035 weight % B, and 0.05 to 0.10 weight % Zr, balance Ni.

35 **[0007]** These and other features, aspects and advantages of the present invention will become better understood with reference to the following drawings, description and claims.

BRIEF DESCRIPTION OF THE DRAWINGS

40 **[0008]**

Figure 1A is a plot showing 0.2% creep and low cycle fatigue (0.65 % strain) data for comparative alloy sample B and for a conventional alloy (Astroloy);

45 Figure 1B is a plot showing 0.2% creep and low cycle fatigue (0.7 % strain) data for alloy sample C of the invention, for comparative sample D and for conventional alloy U720 LI; and

Figure 1C is a plot showing 0.2% creep and low cycle fatigue (0.9 % strain) data for alloy sample C of the invention, for comparative sample D and for conventional alloy U720 LI.

50 DETAILED DESCRIPTION OF THE INVENTION

[0009] The following detailed description is of the best currently contemplated modes of carrying out the invention. The scope of the invention is best defined by the appended claims.

55 **[0010]** The present invention provides nickel based superalloy compositions useful for forming components for gas turbine engines, such as compressor disks, turbine disks, disk seal plates and spacers. The superalloy compositions of the present invention differ from prior art nickel based superalloys (see, e.g., U.S. 6,521,175 B1 to Mourer, et al.) in that alloys of the invention, *inter alia*, contain tungsten (W) at concentrations greater than 5.1% by weight.

[0011] Compositions of the present invention exhibit fatigue crack initiation life at intermediate temperatures 260°C to

649° C (500 to 1200° F) that is higher by about an order of magnitude as compared with previously disclosed superalloy compositions. Alloys of the present invention have superior low cycle fatigue (LCF) properties as compared with previously disclosed nickel based superalloys. For example, alloys of the present invention may have LCF life in excess of 470,000 cycles at 593 °C (1100° F) and 0.7 % strain. Additionally, compositions of the present invention have superior dwell crack growth resistance at higher temperatures of 649 to 788° C (1200 to 1450° F), as compared with previously disclosed compositions. Alloys of the present invention may exhibit 0.2% creep values greater than 400 hours at 704° C (1300° F) and 100 ksi, and greater than 50 hours at 788° C (1450° F), and 65 ksi.

[0012] Alloy compositions of the present invention may be suitable for forming gas turbine engine components, such as turbine discs. Alloy compositions of the present invention enable turbine disk rim operating temperatures in excess of 760° C (1400° F), while providing a level of fatigue crack initiation resistance at disk bore temperatures (typically 260 to 593° C/500 to 1100° F) at least equivalent to the highest known level of fatigue crack initiation resistance attainable in previously disclosed alloys having much lower high temperature capability as compared with alloys of the invention.

[0013] Commonly assigned US Patent No. 6,468,368 B1 to Merrick, et al., and commonly assigned US Patent Application Publication No. 2003/0079809 A1 also to Merrick, et al. disclose a nickel based superalloy which contains 4.5 to 7.5 weight % (tungsten + rhenium).

[0014] Alloy compositions disclosed by Merrick et al. (US 6,468,368) exhibit strength and creep resistance as well as stability at high temperatures eg 649 to 816° C (1200 to 1500° F) (see data for the sample designated as Alloy 1, Figures 1B-C). As will be appreciated, nickel based superalloys which have similar, or the same, components may have markedly different and unexpected properties according to the proportion of the various components. For example, the proportion of alloy components such as W, Nb, Mo, Co, and Ta can have a major impact on the strength, creep resistance, and crack initiation resistance of the alloy. Applicants have now identified compositions having superior dwell crack growth resistance at higher temperatures of 649-788° C (1200 to 1450° F), and a high level of fatigue crack initiation resistance at disk bore temperatures (typically 260-593 °C/500 to 1100° F), as compared with previously disclosed compositions.

[0015] Superalloy compositions of the present invention may be produced by inert gas atomization, and consolidated by hot isostatic pressing (HIP), or hot compaction. The material can be used in HIP form, or may be extruded for forging stock to make isothermally forged turbine engine disks or other components. Such production processes are well known in the art.

[0016] According to the invention, which may be designated Alloy 1.2, a nickel based superalloy composition may comprise from about 16.75 to 17.25 weight % Co, 10.5 to 11.2 weight % Cr, 2.4 to 2.7 weight % Mo, 5.1 to 5.5 weight % W, 3.4 to 3.8 weight % Al, 3.6 to 4.0 weight % Ti, 1.3 to 1.7 weight % Ta, 0.85 to 1.15 weight % Nb, 0.02 to 0.04 weight % C, 0.025 to 0.035 weight % B, and 0.05 to 0.10 weight % Zr, balance Ni. The nickel based superalloy composition designated Alloy 1.2 may exhibit a LCF life at 1100° F, R = 0, 0.7 % strain, of greater than about 470,000 cycles. Alloy 1.2 may further exhibit a time for 0.2% creep, at 704° C (1300° F) and 100 ksi, of greater than 400 hours, in fine grain form.

[0017] As a comparative example, a nickel based superalloy composition, which may be designated Alloy 1.1, may comprise from about 17.7 to 18.5 weight % Co, 10.0 to 10.8 weight % Cr, 2.3 to 2.7 weight % Mo, 4.5 to 5.0 weight % W, 3.4 to 3.8 weight % Al, 3.6 to 4.0 weight % Ti, 1.3 to 1.7 weight % Ta, 0.80 to 1.20 weight % Nb, 0.02 to 0.04 weight % C, 0.025 to 0.035 weight % B, and 0.05 to 0.10 weight % Zr, balance Ni. The nickel based superalloy composition designated Alloy 1.1 may exhibit a LCF life at 800° F, R = -1, 0.65 % strain, of greater than about 260,000 cycles.

[0018] Alloy 1.2 has increased high temperature creep and crack growth resistance capability, as compared with Alloy 1.1. The composition and performance characteristics of a nickel based comparative superalloy designated Sample D (Alloy 1.3), which is intermediate between Alloy 1.1 and Alloy 1.2 with respect to its content of C, Cr, Co, Nb, Al, and B, is described in Example 3.

[0019] A comparative alloy having a composition intermediate between those of Alloys 1.1 and 1.2 (e.g., Alloy 1.3 (Example 3)) may comprise about 17.4 weight % Co, about 11.0 weight % Cr, about 2.56 weight % Mo, about 5.5 weight % W, about 3.64 weight % Al, about 3.8 weight % Ti, about 1.47 weight % Ta, about 0.94 weight % Nb, about 0.03 weight % C, about 0.03 weight % B, and about 0.1 weight % Zr, balance Ni. A superalloy such as Alloy 1.3 may exhibit a LCF life, at 1100° F and 0.7 % strain, of greater than about 200,000 cycles.

[0020] In one embodiment, nickel based superalloy compositions of the present invention may be formed by the Powder Metallurgy (P/M) route, for example, as described in commonly assigned US Patent No. 6,468,368 B1 to Merrick, et al.

[0021] The nickel based superalloy compositions of the present invention may optionally further include rhenium in an amount from 0 to 2.0 weight %, and usually at or near 0 weight %. Generally, rhenium may have little or no effect on superalloy properties, but may result in a slight enhancement of creep performance.

[0022] The nickel based superalloy compositions of the present invention may optionally further include hafnium in an amount from 0 to 1.0 weight %, although amounts greater than 0% may have a negative impact on LCF properties, as seen in some prior art superalloys. Additional elements, such as magnesium (up to 0.1 weight %), may also be added to superalloy compositions of the invention, typically with no substantial effect on properties.

EXAMPLESExample 1

[0023] A comparative alloy designated Sample B (Alloy 1.1 B) was prepared having the following composition expressed as weight %: 18.2 % Co, 10.5 % Cr, 2.65 % Mo, 4.8 % W, 3.57 % Al, 3.86 % Ti, 1.65 % Ta, 0.95 % Nb, 0.027 % C, 0.028 % B, and 0.07 % Zr, balance Ni. A conventional alloy (Astroloy) was also prepared, and the fatigue and creep characteristics of HIP processed Sample B and Astroloy were compared. For both the Astroloy and Sample B alloy, 270 mesh powder was used. Both the Astroloy and Sample B were supersolvus HIP processed at about 1213° C (2215° F), and solution treated to yield a grain size of ASTM 7 to 8. The cooling rate was about 24° C (75° F) per minute from solution treatment temperature for both Astroloy and Sample B.

[0024] The data for LCF life at 427° C (800° F), R = -1, 0.65% strain, and time for 0.2 % creep at 788° C (1450° F) 65 ksi for conventional Astroloy and Sample B of the invention are shown in Figure 1A. Under these conditions the conventional material, Astroloy, had a LCF of 166,810 cycles. In comparison, Sample B (Alloy 1.1B) had a LCF of 266,154 cycles. Similarly, the conventional material, Astroloy, showed a time for 0.2 % creep at 788° C (1450° F) and 65 ksi of five (5) hours. In comparison, Sample B (Alloy 1.1B) exhibited a time for 0.2 % creep at 788° C (1450° F) and 65 ksi of 85 hours. The data from Figure 1A is tabulated below (Table 1).

Table 1. LCF and 0.2% Creep Values for Sample B and PM Astroloy

"Alloy Material"	Time (hours) for 0.2% Creep (788° C/1450° F, 65 ksi)	LCF Life (cycles) (427° C/800° F, R = -1, 0.65% strain)
Sample B	85	266,154
PM Astroloy ¹	5	166,810
¹ conventional superalloy		

Example 2

[0025] Sample A (Alloy 1.1A) was prepared having the following composition expressed as weight %: 17.8 % Co, 10.5 % Cr, 2.6 % Mo, 5.0 % W, 3.58 % Al, 3.9 % Ti, 1.47 % Ta, 1.03 % Nb, 0.028 % C, 0.028 % B, and 0.10 % Zr, balance Ni. The fatigue and creep characteristics of HIP processed Sample A were generally similar to those of HIP processed Sample B as described hereinabove (Example 1 and Figure 1A).

Example 3

[0026] An alloy of the invention designated Sample C (Alloy 1.2C) was prepared having the following composition expressed as weight %: 16.9 % Co, 11.1 % Cr, 2.55 % Mo, 5.5 % W, 3.79 % Al, 3.97 % Ti, 1.57 % Ta, 0.91 % Nb, 0.033 % C, 0.035 % B, and 0.09 % Zr, balance Ni. Sample C was made from 270 mesh powder, hot compacted, extruded, and isothermally forged. The solution treatment was subsolvus solution treated to yield a grain size of ASTM 11-12. The cooling rate from solution temperature was about 54° C (130° F) per minute.

[0027] Sample D (Alloy 1.3), was prepared having the following composition expressed as weight %: 17.4 % Co, 11.0 % Cr, 2.56 % Mo, 5.5 % W, 3.64 % Al, 3.8 % Ti, 1.47 % Ta, 0.94 % Nb, 0.03 % C, 0.03 % B, and 0.1 % Zr, balance Ni. Sample D was made from 270 mesh powder, hot compacted, extruded and isothermally forged. The solution treatment was subsolvus to yield a grain size of ASTM 10-11. The cooling rate from solution temperature was about 260° C (500° F) per minute.

[0028] The data for low cycle fatigue (LCF) life at 593° C (1100° F), R = 0, 0.7% strain, and time for 0.2 % creep at 704° C (1300° F), 100 ksi, for Samples C of the invention and comparative example D are shown in Figure 1B. For comparison, conventional alloy U720 LI was tested under the same conditions. Alloy 1 represents an alloy composition according to commonly assigned US Patent No. 6,468,368 B1 to Merrick et al. Sample C of the invention and comparative example D had a LCF life of 472,876 cycles and 205,610 cycles, respectively; and a time for 0.2 % creep at 704° C (1300° F) and 100 ksi of 432 hours and 450 hours, respectively.

[0029] Under these conditions, LCF values for Samples C and D, respectively, are almost five times (5X) and more than twice (>2X) the LCF value for conventional alloy U720 LI. Time for 0.2 % creep for Samples C and D is about two (2) orders of magnitude greater than that for conventional alloy 720. It can also be seen from Figure 1B that under the specified test conditions, LCF values and time for 0.2 % creep for Samples C and D are at least several fold higher than those for Alloy 1.

[0030] Data for LCF life at 593° C (1100° F), R = 0, 0.9% strain for Samples C and D (Example 3) are shown in Figure 1C. Data for the conventional alloy, U720 LI, and for Alloy 1, tested under the same conditions, are included for comparison. It can be seen from Figure 1C that under the specified test conditions, LCF values and time for 0.2 % creep for Samples C and D are at least several fold higher than those for alloy U720 LI and Alloy 1. The data from Figures 1B and 1C are tabulated below (Table 2).

Table 2. LCF and 0.2% Creep Values for Various Superalloys

Alloy Material	Time (hours) for 0.2% Creep (704° C/1300° F, 100 ksi)	LCF Life (cycles) 0, (593° C/1100° F, R = 0.7% strain)	LCF Life (cycles) (593° C/1100° F, R = 0, 0.9% strain)
Sample C	432	472,876	221,776
Sample D	450	205,610	61,860
U720 LI ²	5	95,911	7,263
Alloy 1 ³	85	66,550	9,850
² conventional superalloy; ³ alloy of Merrick et al. (US 6,468,368).			

Claims

1. A nickel based superalloy composition, comprising: 16.75 to 17.25 weight % Co, 10.5 to 11.2 weight % Cr, 2.4 to 2.7 weight % Mo, 5.1 to 5.5 weight % W, 3.4 to 3.8 weight % Al, 3.6 to 4.0 weight % Ti, 1.3 to 1.7 weight % Ta, 0.85 to 1.15 weight % Nb, 0.02 to 0.04 weight % C, 0.025 to 0.035 weight % B, 0.05 to 0.10 weight % Zr, 0-2 weight % Re, and 0-1 weight % Hf, balance Ni and any incidental impurities.
2. The nickel based superalloy composition of claim 1, comprising: about 16.9 weight % Co, 11.1 weight % Cr, 2.55 weight % Mo, 5.5 weight % W, 3.79 weight % Al, 3.97 weight % Ti, 1.57 weight % Ta, 0.91 weight % Nb, 0.033 weight % C, 0.035 weight % B, and 0.09 weight % Zr.
3. The nickel based superalloy composition of claim 5, wherein said superalloy exhibits a LCF life, at 1100° F, R = 0, 0.7 % strain, of greater than about 470,000 cycles.
4. A gas turbine engine component formed from the nickel based superalloy composition of any one or more of claims 1-3.
5. The nickel based superalloy composition of claim 1, further comprising at least one element selected from the group consisting of up to 2 weight % Re, up to 1.0 weight % Hf, and up to 0.1 weight % Mg.

Patentansprüche

1. Nickelbasierte Superlegierungszusammensetzung, umfassend: 16,75 bis 17,25 Gew.-% Co, 10,5 bis 11,2 Gew.-% Cr, 2,4 bis 2,7 Gew.-% Mo, 5,1 bis 5,5 Gew.-% W, 3,4 bis 3,8 Gew.-% Al, 3,6 bis 4,0 Gew.-% Ti, 1,3 bis 1,7 Gew.-% Ta, 0,85 bis 1,15 Gew.-% Nb, 0,02 bis 0,04 Gew.-% C, 0,025 bis 0,035 Gew.-% B, 0,05 bis 0,10 Gew.-% Zr, 0 bis 2 Gew.-% Re, und 0 bis 1 Gew.-% Hf, Rest-Ni und jede zufällige Verunreinigung.
2. Nickelbasierte Superlegierungszusammensetzung nach Anspruch 1, umfassend:
etwa 16,9 Gew.-% Co, 11,1 Gew.-% Cr, 2,55 Gew.-% Mo, 5,5 Gew.-% W, 3,79 Gew.-% Al, 3,97 Gew.-% Ti, 1,57 Gew.-% Ta, 0,91 Gew.-% Nb, 0,033 Gew.-% C, 0,035 Gew.-% B und 0,09 Gew.-% Zr.
3. Nickelbasierte Superlegierungszusammensetzung nach Anspruch 2, wobei die Superlegierung eine LCF-Lebensdauer bei 1100° F von R = 0,07 % Beanspruchung von mehr als etwa 470.000 Zyklen zeigt.
4. Gasturbinenmotorkomponente aus der nickelbasierten Superlegierungszusammensetzung nach einem der Ansprüche 1 bis 3.

5. Nickelbasierte Superlegierungszusammensetzung nach Anspruch 1, ferner umfassend mindestens ein Element, das ausgewählt ist aus der Gruppe, bestehend aus bis zu 2 Gew.-% Re, bis zu 1,0 Gew.-% Hf und bis zu 0,1 Gew.-% Mg.

5

Revendications

10

1. Composition de superalliage à base de nickel, comprenant de 16,75 à 17,25 % en poids de Co, de 10,5 à 11,2 % en poids de Cr, de 2,4 à 2,7 % en poids de Mo, de 5,1 à 5,5 % en poids de W, de 3,4 à 3,8 % en poids d'Al, de 3,6 à 4,0 % en poids de Ti, de 1,3 à 1,7 % en poids de Ta, de 0,85 à 1,15 % en poids de Nb, de 0,02 à 0,04 % en poids de C, de 0,025 à 0,035 % en poids de B, de 0,05 à 0,10 % en poids de Zr, de 0 à 2 % en poids de Re et de 0 à 1 % en poids de Hf, le solde étant le Ni et les impuretés inévitables.

15

2. Composition de superalliage à base de nickel selon la revendication 1, comprenant environ 16,9 % en poids de Co, 11,1 % en poids de Cr, 2,55 % en poids de Mo, 5,5 % en poids de W, 3,79 % en poids d'Al, 3,97 % en poids de Ti, 1,57 % en poids de Ta, 0,91 % en poids de Nb, 0,033 % en poids de C, 0,035 % en poids de B et 0,09 % en poids de Zr.

20

3. Composition de superalliage à base de nickel selon la revendication 2, dans lequel ledit superalliage présente à 1 100°F, R = 0 et contrainte de 0,7 % une durée de vie LCF supérieure à environ 470 000 cycles.

4. Composant de moteur à turbine à gaz formé de la composition de superalliage à base de nickel selon l'une quelconque des revendications 1 à 3.

25

5. Composition de superalliage à base de nickel selon la revendication 1, comprenant en outre au moins un élément sélectionné dans l'ensemble constitué de jusque 2 % en poids de Re, de jusque 1,0 % en poids de Hf et de jusque 0,1 % en poids de Mg.

30

35

40

45

50

55

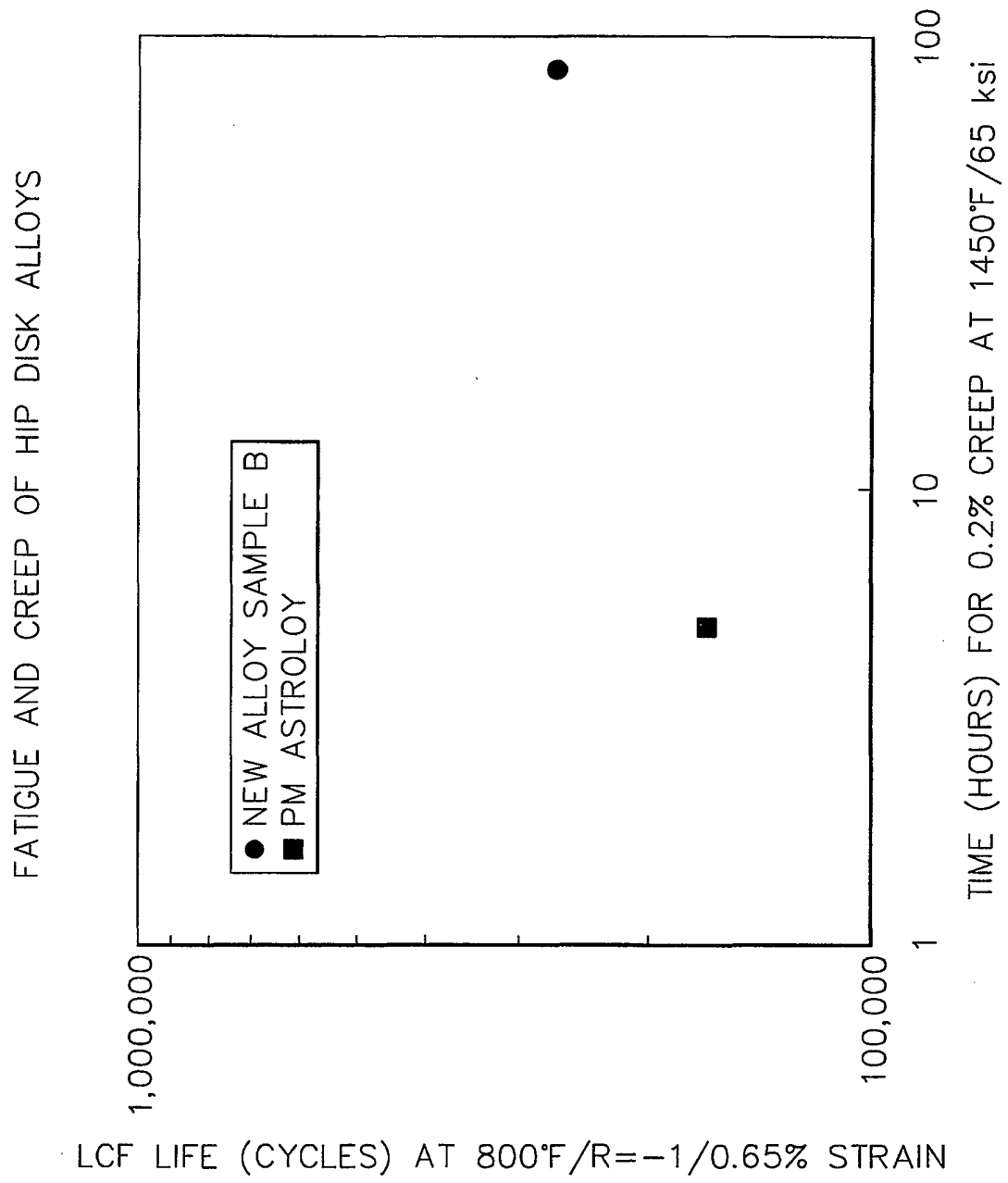


FIG. 1A

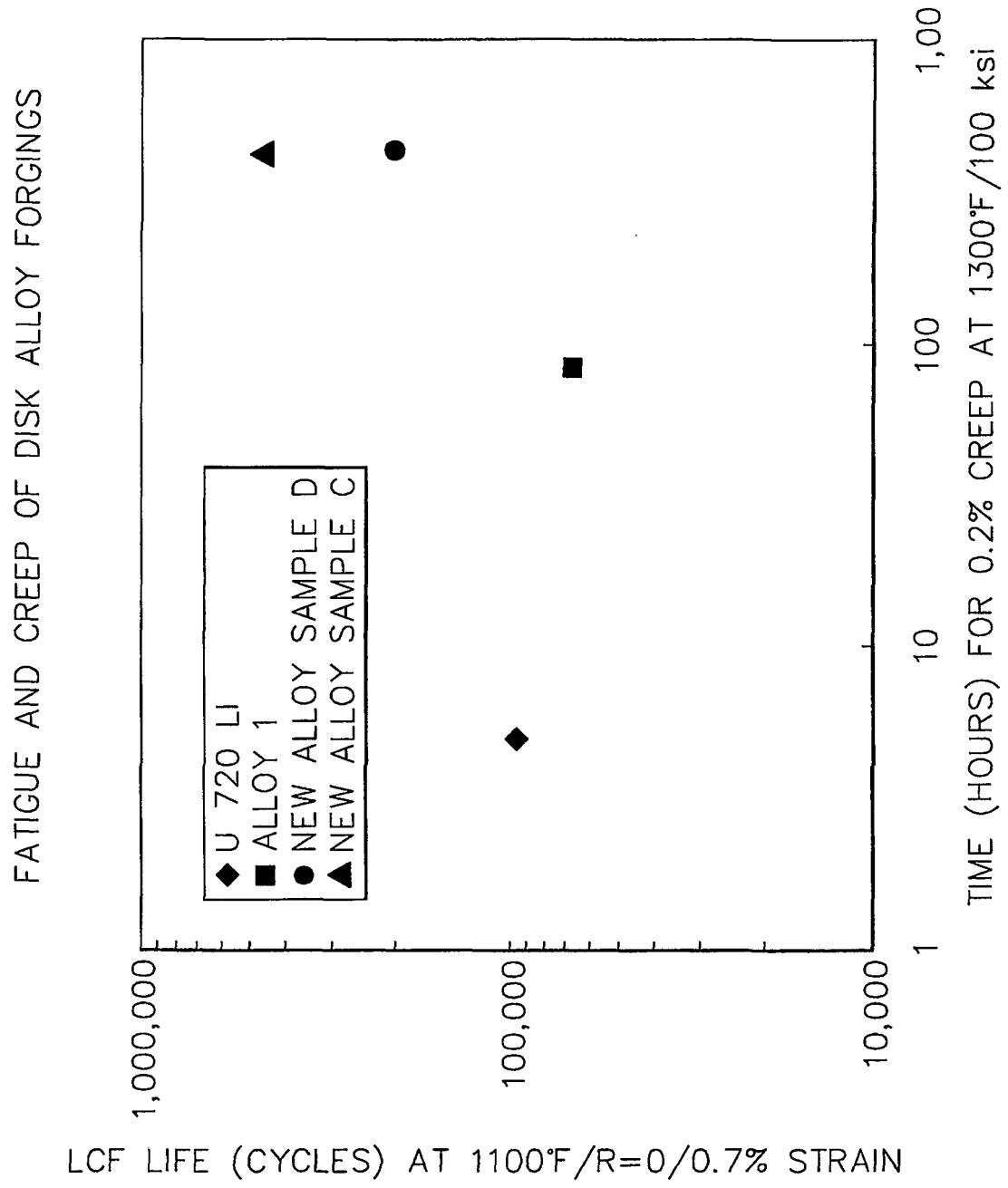


FIG. 1B

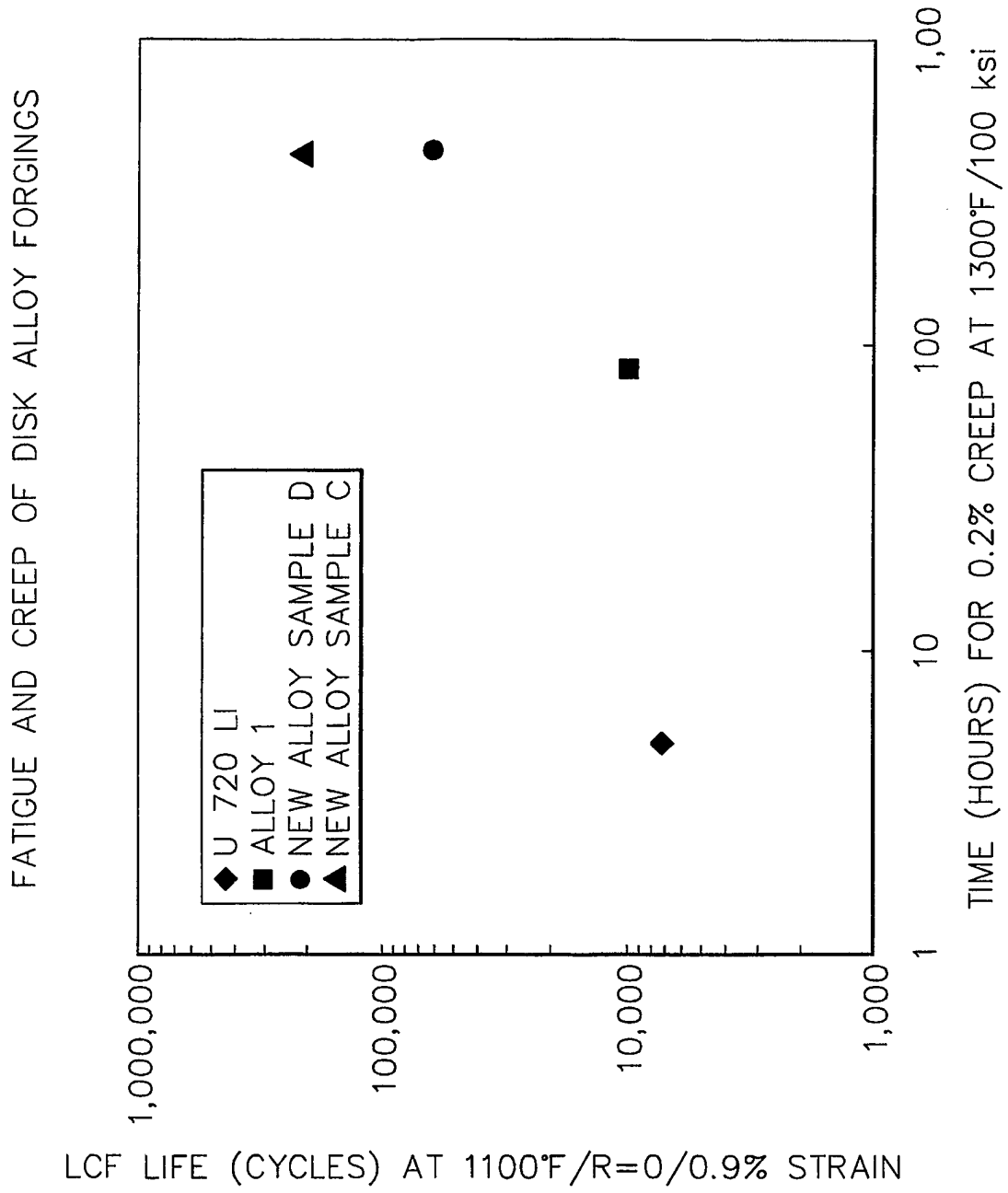


FIG. 1C

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- US 6521175 B1, Mourer [0004] [0010]
- US 6468368 B1, Merrick [0013] [0020] [0028]
- US 20030079809 A1, Merrick [0013]
- US 6468368 B, Merrick [0014] [0030]